Pilot-Plant Concentration of Cheese Whey by Reverse Osmosis

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SUMMARY

Pilot plant studies on conditions affecting concentration of cheese whey by reverse osmosis are reported. Variables include pressure, membrane porosity, feed rate, temperature and pH. As expected, concentration rates increased as pressure and/or membrane porosity increased. However, maximum pressure was limited because of possible compaction of the membrane. Less than 1% of the total solids was lost in the permeate through the tighter membranes and 3.7% was lost through the loose membrane. Results indicate that concentration to 4:1 will be practical but higher levels will be uneconomical and can cause clogging problems from an accumulation of viscous materials and insoluble solids.

INTRODUCTION

The annual production of some 22 billion lbs of whey, a byproduct of cheese manufacture, presents a major water pollution problem. Whey contains half the solids of milk and is rich in vitamins, amino acids, lactose, and soluble protein. Though much of it is used for animal feed, lactose production, or as a food additive, at least half is creating a waste disposal problem.

A recent analysis of disposal methods of 590 cheese factories in Wisconsin (Groves et al., 1965) reveals that over 50% of the plants paid about 1¢ per cwt for disposal of the whey as waste or sewage, 25% paid about 2¢ per cwt to processors to dispose of it for them, and 15% gave the whey to farmers at no cost. The remaining 9 to 10% of the plants either sold or processed their own. Unfortunately, whey is highly perishable and must be concentrated and/or dried for future use. Much of the production is in plants too small to afford the installation and operation of conventional concentrating or drying equipment.

Reverse osmosis, the selective removal of water from a solution through a semi-permeable membrane, has the potential for being an effective low-cost method for concentrating and fractionating whey. Reid et al. (1959) showed cellulose acetate to be an effective material for water desalination, but the early membranes had low permeabilities until Loeb et al. (1960) improved them by altering the casting technique. Therefore, reverse osmosis gained attention as a method for desalination of water and has been successfully used for concentration of maple sap, fruit juice and cheese whey (Lowe et al., 1968; Marshall et al., 1968; McDonough, 1968; Merson et al., 1968; Willits et al., 1967).

This paper reports results of pilot plant studies designed to determine optimum conditions and factors affecting concentration of cheese whey by reverse osmosis. Cost estimates are given based on the results.

EXPERIMENTAL PROCEDURE

Whey. Cheddar cheese whey was obtained from the Dairy Products Laboratory pilot plant at the U.S. Department of Agriculture's Research Center at Beltsville, Maryland. Cottage cheese whey was obtained from a local dairy. Both types were usually clarified in a centrifugal clarifier, pasteurized by the HTST method, and held at 40°F for no longer than three days prior to concentration. Some lots were processed without pasteurization or clarification to determine the effects of "fines" on concentration and clogging.

Equipment. The unit used for these tests was the Model II "Osmotik" separator developed by Havens Industries. Tubular type modules are the fundamental part of the unit. A description of the basic types of modules has been given elsewhere (McDonough, 1968). Each module of the unit tested contains seven pressure tubes imbedded in an inboard fitting at the ends. The tubes, which are eight ft long and have an ID of ½ in., are of fiberglass which provides backing for the integral formed cellulose acetate membrane layer. Each tube contains 0.93 ft² of active membrane surface; thus a seven tube module contains 6.5 ft². The tubes are connected with "U" turn connectors at each end so each module represents 56 ft of membrane lined tubing. A shroud surrounds the seven tubes and collects the permeate water filtering through.

The complete mobile unit consists of a frame containing 48 modules, 16 each of types 3A, 4A, and 5A. These correspond to salt rejections (based on 5000 ppm NaCl) of 75%, 91% and 95% respectively. Thus each bank of 16 modules contains approximately 104 ft² of membrane surface. Valves are arranged so that each bank can be operated independently or in series with the remaining banks. Pressure was provided by a variable speed Moyno model 9P3SSQ positive displacement pump, capable of producing up to 800 psi.

Procedure. Prior to initiating whey concentration trials, permeation rates were determined for each type bank using 75°F water supplied at varying feed rates and pressures. These data were periodically compared to similar tests during the course of the study in order to detect changes in permeability due to clogging or compaction.

Each experiment was conducted on each membrane type on successive days, using 80 to 100 gal of compositely mixed whey per test. The whey was adjusted to 75°F in a hollow jacketed stainless steel vat and maintained at that temperature by circulating water through the jacket. Experimental lots of Cheddar and Cottage whey, both clarified and unclarified, were concentrated using pressures of 600, 700, and 800 psi at feed velocities of 2 to 5 gal/min. Whey was recirculated through the system until flux rates slowed to a negligible level. The permeate was collected in a separate vat.

The modules were cleaned by pumping tap water through the system until the dissolved solids of the permeate, as measured on a Myron L-Delux Dissolved Solids Meter, were as low as the original tap water. This was occasionally followed by a 15 ppm chlorine solution.

Analytical. Samples of the feed,

three days. After each use, the equipment was thoroughly flushed with warm tap water. Although analysis of the water remaining in the system showed the absence of nutrient material, foul odors and a pinkish green discoloration developed in three to five days.

It is possible that the modular material can serve as a nutrient for some bacteria; thus, cleaning must be supplemented with bactericidal agents which do not damage the membranes. Some agents, such as hydrogen peroxide cause hydrolysis and cannot be used. A study is needed to assess the potential of other chemicals.

Economics. A cost analysis for the process is difficult at this stage of development, especially when based on small scale tests. Several studies have been made on the economics of reverse osmosis as a process for desalination of water, but these studies have no significance for the dairy industry. The main areas influencing economics of whey concentration are as follows:

1. Cost of reverse osmosis equipment. The costs of pumps, piping, instrumentation, etc., are well known and changes in technology are not expected to create savings here. Modules, the basic part of the unit, will be improved and cost reductions of up to 50% may be expected. A major part of the present cost of reverse osmosis equipment is for the technology involved. Mass production would undoubtedly lower costs even more. At the present time a completely equipped mobile unit with 300 ft² of membrane will cost approximately $8,000. A comparable unit containing 900 ft² of membrane would be about $17,000.

2. Life of the membrane. The modules or membranes within the modules are the only parts expected to need replacement. Reasonable life of the membranes is essential for success. Knowledge of the effects of whey on deterioration of the membranes is lacking but some manufacturers are predicting a life of two years.

3. Flux rates. This is the area where improvement can contribute most to lowering of costs. It is likely that improvements in technology of membrane casting will increase flux rates, resulting in substantial reductions in processing costs.

4. Labor. Assuming trouble-free operation, labor will be a minor cost. Close supervision of the equipment is unnecessary and it can be fully automated.

5. Power costs. Since no phase changes are needed for reverse osmosis, the only energy requirement is the electricity needed to drive the pump. Power requirements to pump 4.5 gal/min at 800 psi through a 900 ft² unit would be about 4.5 kw per hr. This can be purchased for about 12¢ at usual electricity rates.

Table 3. Cost data for concentration of whey by reverse osmosis.[1]

Concentration rate		Cost[2]	
	(lb per yr)	(per 1000 lb whey)	(per lb H_2O removed)
2:1	17,690,400	$0.34	$.00073
3:1	9,752,400	.63	.0010
4:1	6,577,200	.94	.0013

[1] Based on type 3A membrane, operated 10 hr day, 300 day year at 800 psi.

[2] Based on an annual expenditure of $3,400.00 for equipment (900 ft² membrane unit at $17,000.00, depreciated in 5 years), $2,500.00 for membrane replacement (based on two-year life) and $300.00 for energy costs.

Based on the above considerations and utilizing concentration rate data from this study, a cost estimate is given in Table 3. It assumes the use of a 900-ft² unit, a 10-hr day and a 300-day year using type 3A membrane at 800 psi. Since the degree of concentration is important in calculating costs, figures are given for concentrations of 2:1, 3:1, and 4:1. Cost of overhead, labor, insurance, etc. are not included.

Table 3 indicates that concentration by reverse osmosis is economically sound. Our estimate of the cost of concentrating to 3–1 is approximately 0.10 cent/lb water removed. Havens Industries calculate a slightly lower cost; they offer equipment on a lease basis for about 0.09 cent/lb water removed when concentrating to over 4–1. The comparable cost to 3–1 would be substantially lower than to 4–1.

These figures indicate a savings over conventional thermal evaporation. A representative of a leading manufacturer of dairy processing equipment (Bauman, A. W., personal communication) estimates a concentration cost to 3–1 of at least 0.1 cent/lb water removed, assuming the use of their smallest unit and processing of twice the volume of whey reported here. He further feels that processing of smaller volumes of whey would be completely uneconomical using thermal methods.

Experience to date indicates that reverse osmosis will be of value for small volume plants, although the degree of concentration will be limited. Problems multiply rapidly as concentration increases beyond about 4:1. Lactose crystallization, accumulation of viscous materials and insoluble solids on the membrane, and increased viscosity effects are some of the problems Marshall et al. (1968) encountered at high concentration in a recent study using a plate and frame type unit. Their recommendations of several steps included more than one clarification. Though no cost estimates were given, it is doubtful that processing to concentrations in excess of 4:1 with present reverse osmosis equipment is economical. Concentration to 30% solids removes over 80% of the water. The value of paying the increased cost to remove an additional 10% of the water is suspect. By limiting the pressure to 800 psi, and by concentrating only to 25 to 30% solids, the process will be feasible. Longer term commercial operation is now required to define the real problems and potentialities.

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Mention of brand or firm names does not constitute an endorsement by the Department of Agriculture over others of a similar nature not mentioned.

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and a point of equilibrium would be reached for a given pressure. Therefore, as the pressure is increased within module strength limits, the greater will be the rate and extent of concentration. Other factors influencing pressure selection are increased cost of high capacity pumps and reduced flux rates because of irreversible compaction of the membranes. Compaction will be discussed in connection with membrane life.

Wiley et al. (1967) have shown the influence of temperature on flux rates. They obtained a twofold increase in permeation rates, using pure water, by increasing the temperature from 18°F to 88°F. Manufacturers have found that higher temperatures lessen the life of the membranes and they suggest a limit of 90°F.

Flow rate, or feed velocity, is of extreme importance for effective osmotic processing. As a layer of feed material travels across the membrane, the water adjacent to the membrane surface passes through, resulting in a concentration gradient with layers of uneven concentration. The boundary layer next to the membrane has a significantly greater osmotic pressure which soon equals the operating pressure of the system, curtailing the flux. The above phenomenon occurs during laminar flow. It can be overcome by pumping feed material through the tubes with sufficient turbulence to overcome boundary layers and minimize the osmotic pressure at the interface. A velocity with a Reynolds number of 4000 to 5000 is required. Though higher flow rates improve permeation, excessive velocity can damage the membranes and is not recommended. A rate of about 2 gal/min appeared to be optimum.

Table 1. Analytical data from reverse osmosis concentration of whey.[1]

Sample	Membrane[2]	Weight	Total solids	Concentration ratio	Titratable acidity	Ash	Nitrogen	Lactose
		(lbs)	(%)			(%)	(%)	(%)
Cheddar whey—A	5A	680	7.31	——	0.16	0.531	0.139	5.20
Concentrate—A	5A	151.1	32.5	4.46	0.85	2.36	0.59	23.3
Permeate—A	5A	528.9	0.07	——	0.07	0.015	0.009	0.037
Cheddar whey—B	4A	680	7.31	——	0.16	0.531	0.139	5.20
Concentrate—B	4A	153.7	31.8	4.35	0.74	2.22	0.59	22.7
Permeate—B	4A	526.3	0.09	——	0.11	0.026	0.007	0.040
Cheddar whey—C	3A	680	7.31	——	0.16	0.531	0.139	5.20
Concentrate—C	3A	153.6	31.4	4.29	0.72	1.83	0.57	22.8
Permeate—C	3A	526.4	0.27	——	0.29	0.13	0.012	0.056
Cottage whey—D	5A	680	6.53	——	0.57	0.607	0.135	4.39
Concentrate—D	5A	141.4	31.4	4.80	2.38	2.88	0.64	20.98
Permeate—D	5A	538.6	0.06	——	0.08	0.017	0.003	0.037
Cottage whey—E	4A	680	6.53	——	0.57	0.607	0.135	4.39
Concentrate—E	4A	142.6	29.6	4.53	2.50	2.80	0.62	20.70
Permeate—E	4A	537.4	0.07	——	0.09	0.023	0.006	0.070
Cottage whey—F	3A	680	6.53	——	0.57	0.607	0.135	4.39
Concentrate—F	3A	132.1	32.6	4.99	2.50	2.55	0.63	22.2
Permeate—F	3A	547.9	0.30	——	0.12	0.18	0.014	0.07

[1] Concentrate at 700 psi with 2 gal per min flow rate.
[2] Manufactured by Havens Industries; corresponds to salt rejection rates of 95.5% for type 5A, 91.1% for 4A and 75% for 3A.

Table 2. Dry weight analysis of concentrate and permeate from whey processed by reverse osmosis.[1]

		Cheddar whey			Cottage whey		
		Membrane type			Membrane type		
		5A	4A	3A	5A	4A	3A
Total solids (lbs)	Whey	49.64	49.64	49.64	44.40	44.40	44.40
	Concentrate	49.37	49.17	48.22	44.08	44.03	42.76
	Permeate	0.37	0.47	1.42	0.32	0.37	1.64
	% Loss	0.74	0.94	2.86	0.72	0.83	3.69
Ash (lbs)	Whey	3.61	3.61	3.61	4.13	4.13	4.13
	Concentrate	3.53	3.47	2.92	4.04	4.06	3.15
	Permeate	0.08	0.14	0.68	0.09	0.12	0.98
	% Loss	2.18	3.79	18.94	2.22	3.00	23.72
Lactose (lbs)	Whey	35.36	35.36	35.36	29.85	29.85	29.85
	Concentrate	35.16	35.15	35.06	29.65	29.65	29.46
	Permeate	0.19	0.21	0.29	0.19	0.19	0.38
	% Loss	0.55	0.59	0.83	0.64	0.66	1.28
Nitrogen (lbs)	Whey	0.945	0.945	0.945	0.918	0.918	0.918
	Concentrate	0.898	0.908	0.882	0.902	0.886	0.842
	Permeate	0.047	0.037	0.063	0.016	0.032	0.076
	% Los	4.97	3.91	6.66	1.74	3.48	7.27

[1] Analysis based on results in Table 1.

Membrane stability. The durability of cellulose acetate membranes is limited; they will deteriorate resulting in increased permeability to both water and dissolved solids (Marshall et al., 1968). Deterioration is caused by both a pressure dependent and a chemical effect.

Lonsdale (1965) showed a high pressure deterioration of the membranes and attributed it to compaction. He believes that the development of additional flow resistance due to compression of the substructure is responsible. In our studies at 800 psi, we encountered a 4% reduction in permeability of water after 6 months of intermittent operation on the 3A membrane. No compaction was evident on the 4A and 5A membranes during the same time. Manufacturers state that the tighter membranes will withstand pressures up to 1500 psi before compaction occurs.

Chemical deterioration results in hydrolysis of the cellulose acetate. Recently, Vos et al. (1966) have studied the hydrolysis rate and have established temperature and pH dependence. They report minimum hydrolysis at pH 4.5 to 5.0 with rapid deterioration occurring above 7.0 or below 2.5. Fortunately, whey is in the acceptable range.

The lifetime of a membrane depends upon the degree of deterioration considered acceptable. Some increase in permeability of solids can occur and still give good results. Vos et al. (1966) calculated that in work with brackish water at 23°C and pH 4.8, the membrane would last several years. No long term trials with whey have been reported but manufacturers estimate a life of 2 years. Reasonable life of the membrane will be critical to the ultimate success of reverse osmosis.

Sanitation. Processing of whey for food use requires attention to sanitation. A major sanitation study was not a part of these tests, but it was evident that such a study would be necessary in the future. No problems were encountered during processing since the use of high feed velocities did not allow bacterial deposits to form. In addition, the pH of Cottage whey generally controls the growth of undesirable organisms. Sweeter Cheddar whey was pasteurized prior to use to prevent acidity from increasing.

Sanitation problems became evident following shutdown for more than

the concentrate, and the composite permeate from each run were analyzed for pH, titratable acidity, total solids by the Mojonnier method, ash by combustion at about 550°C, lactose by a modification of the method of Folin et al. (1918), total nitrogen by the Kjeldahl procedure, and non-protein nitrogen by precipitation with trichloroacetic acid.

RESULTS AND DISCUSSION

Flux rates. Figure 1 shows changes in flux rates through a porous membrane (3A) and a tight membrane (5A) at pressures of 600 and 800 psi. As concentration increases, flux rate differences between membranes become very small. The influence of membrane type and pressure is evident but was not as great after removal of the first 50% of water. The general effect of the variables was predictable; i.e., the higher pressure and the looser membrane gave greater average flux rates. Pressures of 600, 700, and 800 psi were tested for each of the three membrane types. Results using the intermediate membrane (4A) and pressure (700 psi) were in-between those reported.

Concentration rates. Fig. 2 shows typical concentration rates of 80 to 100 gal lots of whey for types 3A and 5A membranes at 600 and 800 psi. As predicted from Figure 1, the greatest difference in rates of concentration, as affected by both pressure and membrane porosity, fell in the low concentration range. Concentration to 15% solids with the 3A membrane averaged 0.77 gal/ft²/hr at 800 psi and 0.51 gal/ft²/hr at 600 psi, a difference of 0.26 gal/ft²/hr. The average to 30% solids was 0.275 and 0.195 gal/ft²/hr at 800 and 600 psi, a difference of only 0.08 gal/ft²/hr.

The general slope of curve for the 5A membrane at 800 psi as compared to that for the 3A at 600 psi is of interest. During concentration to 2:1, there was little difference; however, beyond 15% solids, pressure became more of a factor than membrane porosity. Thus, from a standpoint of concentration rate and purity of the permeate, a combination of 800 psi with the tight membrane would be preferable to 600 psi on the loose one.

Rejection results. Tables 1 and 2 compare compositions of Cheddar and Cottage wheys, concentrates, and permeates from 3A, 4A, and 5A membranes at 700 psi. Differences in the types of whey did not appear to influence separation and concentration characteristics of the membrane. A possible exception may be the type 3A which allowed a slightly greater loss of total solids, ash, and lactose from the Cottage whey. This could probably be expected by the increased degree of ionization in the acid whey.

Membrane types 4A and 5A were very similar in all properties and had good rejection. Losses in the permeate were less than 1% of the total solids, 2 to 4% of the salts, 0.5 to 0.6% of the lactose, and 2 to 5% of the nitrogen. Attempts to precipitate protein from the permeate with trichloroacetic acid failed; thus the nitrogen lost was all non-protein nitrogen such as urea, ammonia salts, and small molecular weight amino acids. The type 3A membrane, being more porous, passed 3.7% of the total solids, 23.7% of the salts, 1.3% of the lactose and 7.3% of the nitrogenous materials.

Process variables. Successful osmotic separation is influenced by several variables, most important of which appear to be pressure, temperature, and flow rate of the feed material. The driving force causing water flow through the membrane is the hydrostatic pressure applied to the feed minus the osmotic pressure difference between the feed and the water removed. Thus, higher pressures are needed to effect separation as osmotic pressure increases with concentration

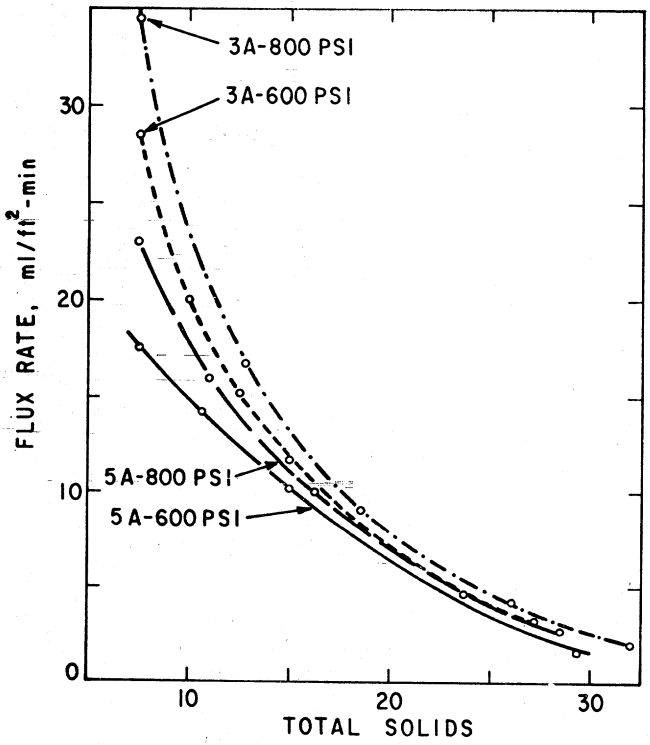

Fig. 1. Effect of whey concentration on flux rates during processing by reverse osmosis. Membranes manufactured by Havens Industries; corresponds to salt rejection rates of 75% for 3A and 95.5% for 5A.

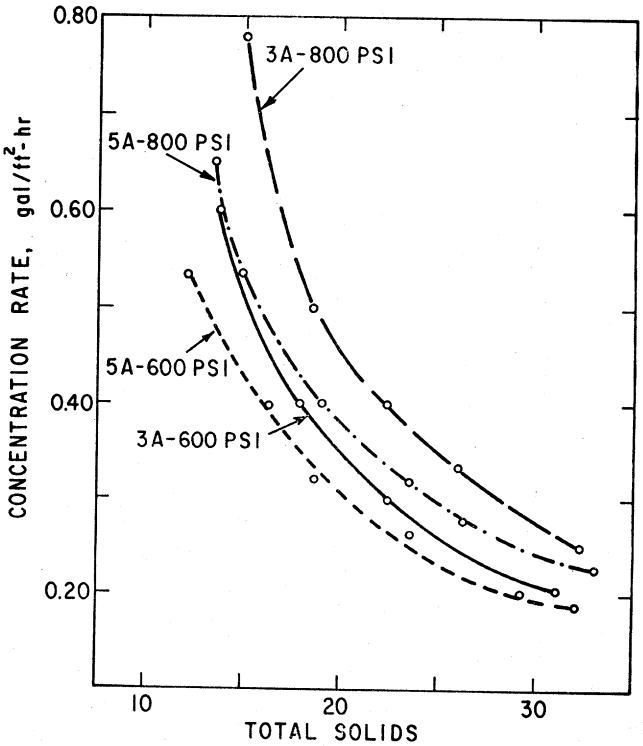

Fig. 2. Influence of membrane type and pressure on average rate of concentration of whey by reverse osmosis. Membranes manufactured by Havens Industries; corresponds to salt rejection rates of 75% for 3A and 95.5% for 5A.